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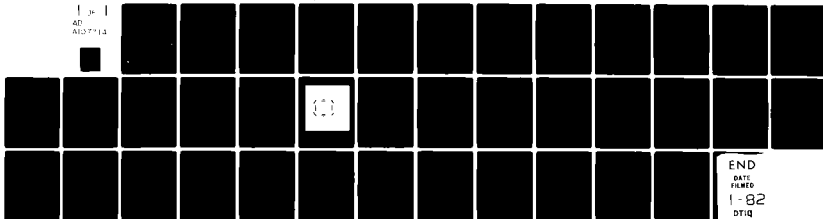
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NEW ENERGETIC MATERIALS - STRUCTURAL CHARACTERIZATION.(U)
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19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Propellants, Elastomers, Explosives, X-ray Diffraction, Electron Diffraction		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The molecular structures of five new materials developed in ONR-sponsored explosive/propellant research are reported. The material 3,3-bis-azidomethyl oxetane (BAMO) was studied in three different forms. Using gas electron diffraction analysis, the molecular structure of the monomer of BAMO in the vapor phase was determined. A cyclic tetramer of BAMO was characterized with X-ray single-crystal diffraction analysis. (CONT'D)		

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Finally, a linear polymer of BAMO was studied as a semi-crystalline fiber using x-rays. Most of the features of the polymer molecular structure were determined from the fiber diffraction pattern.

The crystal and molecular structure of a tetra-nitro compound, 4,7-bis-dinitrofluoromethyl-1,3-dioxepane, is reported; this compound is a newly synthesized monomer developed during research on the synthesis of nitro-polymers. The crystal and molecular structure of bis(5-amino-1,2,4-oxadiazole) is also reported. It is an unusually stable synthetic intermediate obtained in research aimed at producing new types of high-density nitro-explosives.

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Office of Naval Research

Progress Report

New Energetic Materials -
Structural Characterization

Covering the period
Oct. 1, 1980 - Sept. 30, 1981

sponsored by the
Energetic Materials Research Program

Investigators: Dr. Richard Gilardi
Dr. Clifford George
Dr. Jerome Karle

Laboratory for the
Structure of Matter
Naval Research Laboratory
Washington, D. C. 20375

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I. Introduction

This report primarily concerns structural studies on three forms of BAMO, which is an apt acronym for the energetic compound, 3,3-bis-azidomethyl oxetane. Although we have experienced minor explosions with two of these materials, the monomer and the polymer, when working with them at elevated temperatures, all are stable at room temperature.

The BAMO polymer is an elastic white solid with moderate tendencies toward crystallinity. It is currently being manufactured, on a trial basis, for use as an elastic stabilizer for solid mixtures of fuels or explosives. It is insensitive to shock and resistant to accidental ignition, but decomposes rapidly at higher temperatures and thus contributes to the energy of the fuel or explosive.

The cyclic tetramer of BAMO is a by-product produced during polymerization. It can be crystallized as clear colorless prisms which showed no tendency to explode or deteriorate when irradiated with x-rays.

The monomer is a low-vapor-pressure liquid at room temperature which rapidly decomposes at higher temperatures (160-170°C). It was studied in the vapor state by gas electron diffraction analysis.

In addition, the report includes brief summaries of single-crystal diffraction structure determinations of two synthetic intermediates produced in ONR sponsored synthetic research on energetic nitro compounds at the Naval Surface Weapons Center and at the Lawrence Livermore Lab.

II. The Electron Diffraction Analysis of 3,3-bis-azidomethyloxetane (BAMO)

The samples of the monomer were provided by Dr. Kurt Baum of Fluorochem Inc. with a stated purity of 99.5% as determined by NMR. The scattering data were collected on the NRL gas electron diffraction apparatus using the sector-microphotometer method, and covered the range 1.95 to 31.45 s, where $s = (4\pi/\lambda) \sin(\theta/2)$. Here λ is the electron wavelength and θ is the angle the scattered electron beam makes with respect to the incident electron beam. The temperature of the gas injection system was maintained at 120°C during the data collection.

A least squares analysis of the experimental molecular intensity data converges for a model whose geometric parameters are listed in Table (1). The experimental molecular intensity curve and the difference curve, expt.-theo., are shown in Fig. (1). In the course of the analysis the possibility was considered that the vapor phase of the monomer could exist in a number of different conformers at room temperature. The model which best fit the experimental data, however, represents the single conformer shown in Fig. (2). This model has C_2 symmetry. It was computed on the basis that all C-H bond lengths are equal, all H-C-H angles are 110°, all C-C bond lengths are equal, and the N-N-N angle is 180°. In addition, the C-N distance was fixed at 1.46 Å, the average value of this bond length determined from the x-ray diffraction analysis of the crystalline tetramer of BAMO. A shrinkage correction

of 0.005 Å was made for the nonbonded N...N distance in each azido group as well as for the nonbonded C...C and C...O distances in the oxetane ring. The 87 distinct interatomic distances in the model make for a considerable overlap of contributors to the peaks in the radial distribution function, Fig. (3), making it rather unfeasible to determine from the experiment the vibrational amplitudes, l_{ij} , for the nonbonded distances. The l_{ij} values used to perform the calculations for Figs. (1 & 3) have been computed from approximate force constants.

The overall fit of the model to the experimental data is quite satisfactory as seen in Figs. (1 & 3), although the possibility of the presence of additional conformers in small amounts, as well as the presence of some thermal decomposition products cannot be ruled out. Additional analysis of the data is underway to refine the values of the structural parameters and to estimate the possible deviations from ideal values for bonds and angles used in the model calculations.

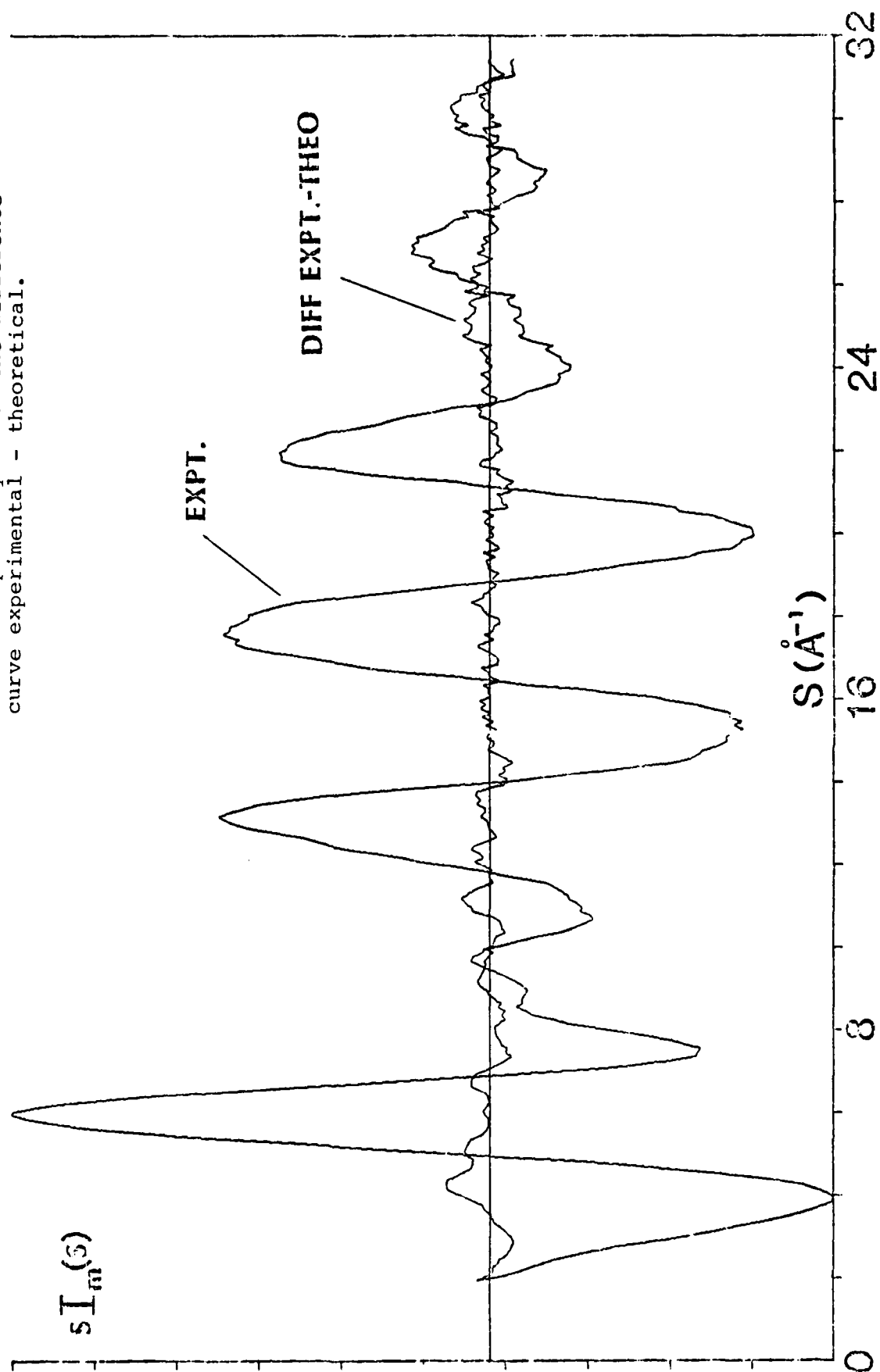
Table 1. Geometric parameters for 3,3-bis-azidomethyloxetane

	r_a (Å)	l_{ij} (Å)	Angle (deg.)
C-H	1.11	0.08	
N ₇ -N ₈	1.14	0.04	
N ₈ -N ₉	1.23	0.04	
C-O	1.47	0.06	
*C-N	1.46	0.05	
C-C	1.54	0.05	
C-C-C			88.5°
C ₅ -C ₁ -C ₆			107.5°
C ₁ -C ₅ -N ₇			111°
C ₅ -N ₇ -N ₈			126.5°
*H-C-H			110°
a) tau			65°
b) omega			90°
c) mu			0°

*assumed

- a) Torsion angle of the methyl groups about the C-C bond
- b) Torsion angle of the azido group about the C-N bond
- c) Angle representing ring pucker in the oxetane ring,
dihedral angle C₂-C₁-C₃-O₄

Fig. (1) Experimental molecular intensity curve for BAMO multiplied by s and the difference curve experimental - theoretical.



BAMO

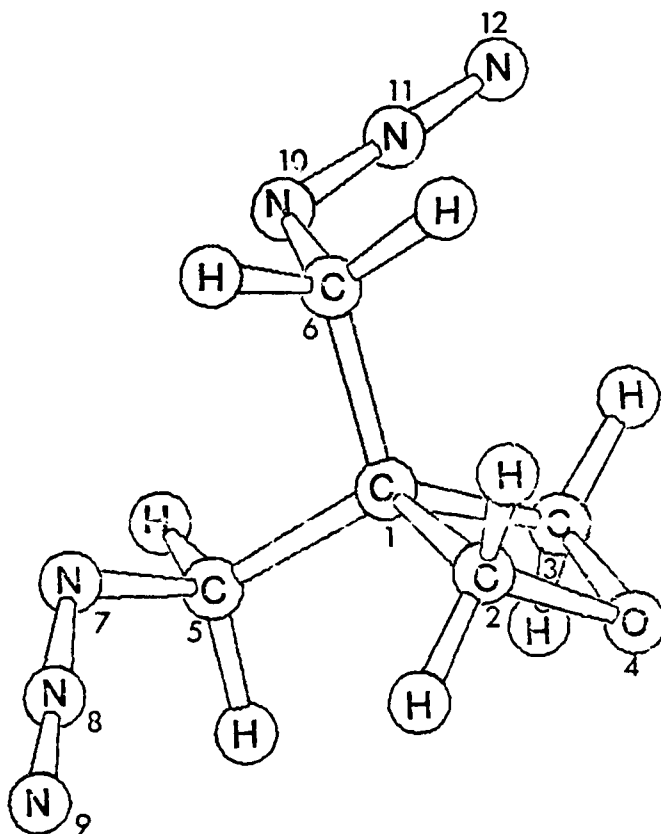
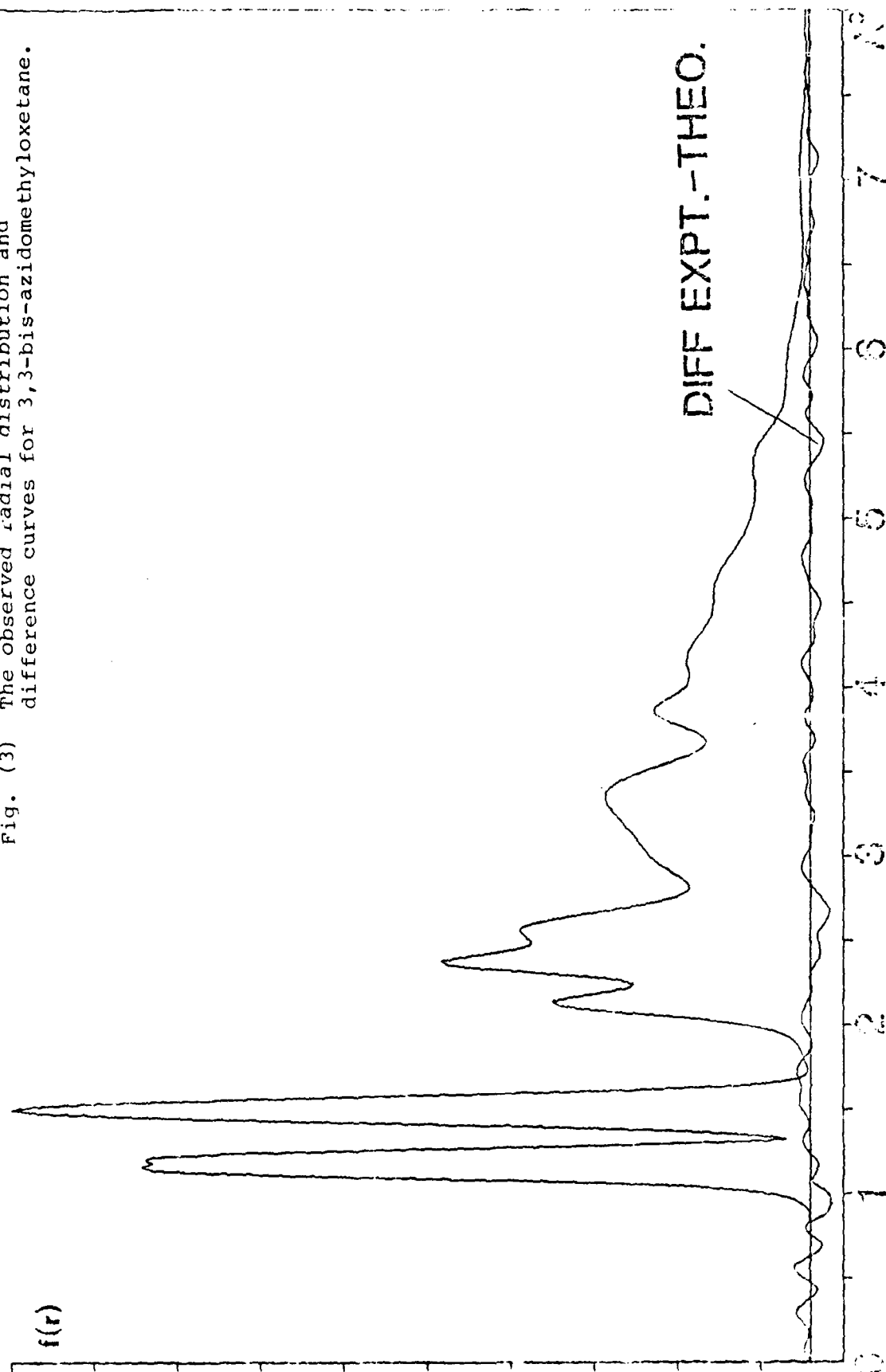


Fig. (2) Computer drawn model of BAMO representing the conformer which fits the experimental diffraction data.

Fig. (3) The observed radial distribution and difference curves for 3,3-bis-azidomethyloxetane.



III. The Crystal Structure of a Cyclic BAMO Tetramer

Oligomers of BAMO may be formed as side products during the course of a polymerization reaction. A crystalline compound was leached from one of the samples of polymeric BAMO provided to us by soaking it in chloroform. This chloroform extract was evaporated and very small crystals (linear dimensions 0.05-0.20 mm.) were formed. At that time, the composition of this side product was unknown, but x-ray diffraction analysis was initiated, assuming the empirical formula would be similar to some multiple of BAMO.

The crystal space group symmetry is Pbcn, orthorhombic, with $a = 12.07$, $b = 15.22$, and $c = 18.18 \text{ \AA}$. The crystal and molecular structure were obtained by using the symbolic addition procedure (Karle and Karle, 1966). As indicated by the title of this section, the molecule is a tetramer of BAMO and thus contains eight methylazido side-chains. In the crystal, the molecule displays only 2-fold symmetry; the left and right halves of the molecule, shown in Fig. 4, are identical. However, the four independent methylazido groups in one half-molecule differ in their conformations, and this indicates that these regions of the molecule are inherently flexible, i.e., in solutions, each methylazido arm can probably shift easily from one conformation to another. In addition, weak "disorder" peaks are visible in the electron density maps near two of the azido side-chains. This indicates that a dynamic libration between alternative energetic minima is occurring to some extent, even in the solid state.

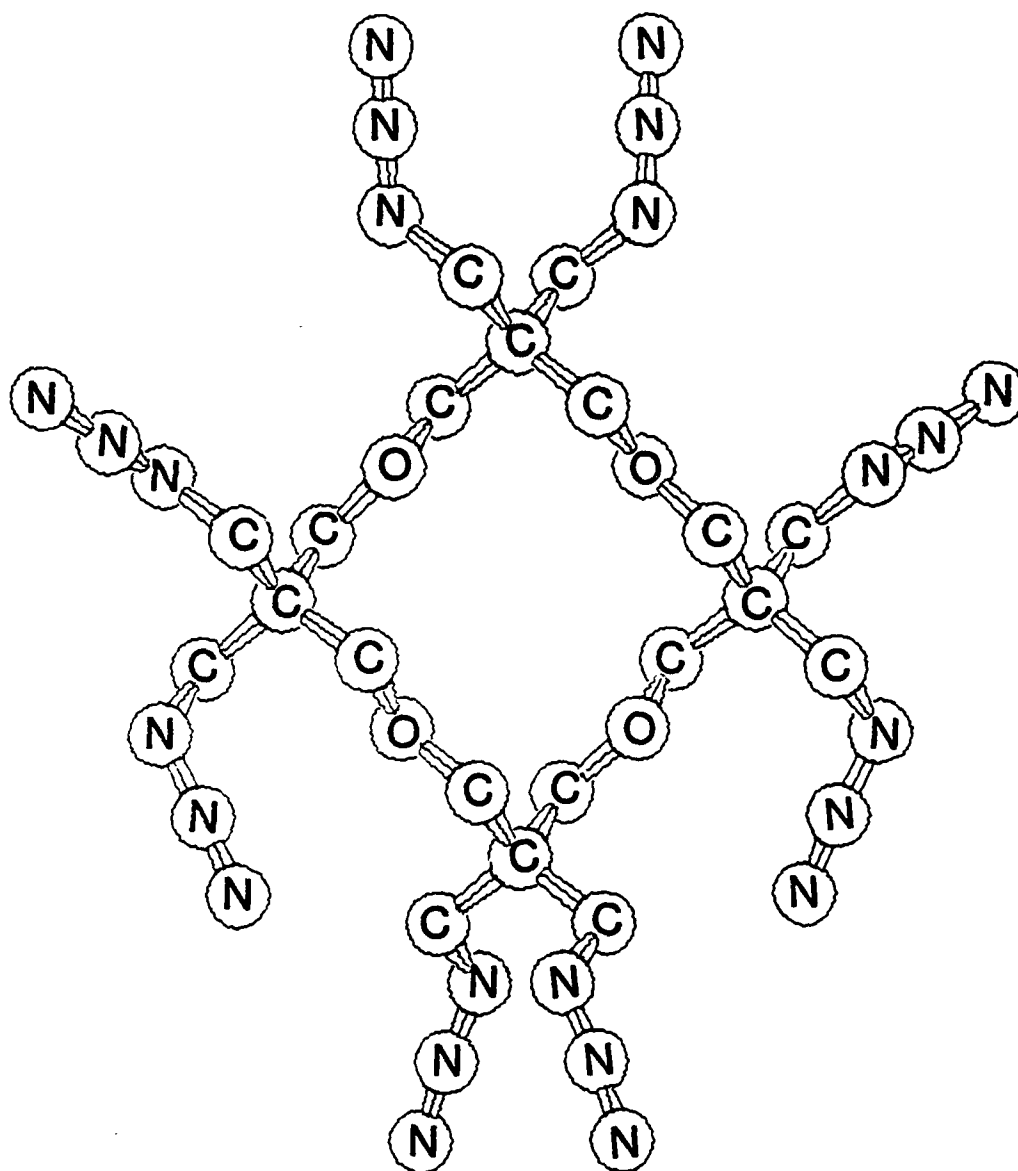


Fig. (4) The molecular structure of cyclic (BAMO)₄. Left and right halves of the molecule are identical in the crystal, related by a vertical two-fold axis.

The bond distances and angles determined for the tetramer (Table 2) provide realistic estimates for the bond distances and angles for the BAMO polymer. The large sixteen-membered ring of the cyclic tetramer contains four planar zig-zag "polyether" segments which are exactly analogous to the backbone linkages in $(\text{BAMO})_n$. The polymer diffraction analysis relies, in its intermediate stages, upon energetic calculations on a segmented polymer model, and these calculations are more likely to be correct if the model is based on a well-characterized small-molecule analog, such as, in this case, the BAMO tetramer.

Table 2. Bond Distances and Angles Obtained for the
BAMO Tetramer through Least-Squares Refinement
of the Model

Type	Distances(Å)	Averages	Type	Angles(°)	Averages
C-O	1.411(7)	1.416	C-O-C	112.1(5)	112.9
	1.419(6)			113.7(5)	
	1.420(7)		O-C-C	108.5(5)	109.1
	1.413(7)			108.5(5)	
1.520(8)	109.3(5)				
1.526(8)	110.0(5)				
C-C	1.519(8)	1.525	C-C-C	109.7(5)	109.7
	1.532(7)			107.2(6)	
	1.509(8)			111.5(6)	
	1.535(8)		110.9(5)		
	1.528(8)		105.9(6)		
	1.531(7)		110.4(4)		
C-N	1.459(9)	1.460	N-C-C	110.0(3)	109.0
	1.440(9)			111.7(5)	
	1.472(10)			110.4(6)	
	1.468(8)			105.9(6)	
N=N	1.254(23)	1.170	N-N-C	110.8(7)	115.7
	1.130(25)			108.9(5)	
	1.175(12)			114.4(7)	
	1.163(7)			114.7(9)	
N≡N	1.159(22)	1.143	N-N-N	116.6(10)	171.5
	1.240(25)			117.1(6)	
	1.120(12)			173.3(10)	
	1.141(8)			172.7(12)	
				169.1(14)	
				170.8(9)	

* The figures in parentheses are the estimated standard deviations in the final digits of each value. Particularly large esd's are noted with asterisks, and are due to azido disorder. Averages are weighted averages (with the weights equal to the reciprocal square of the esd's).

IV. The Crystal and Molecular Structure of the Energetic Polymer, $(\text{BAMO})_n$

Polymers of BAMO show a tendency to crystallize even without special treatment such as annealing or stretching. The initial samples of $(\text{BAMO})_n$ investigated were rubbery cross-linked polymers which contained, besides BAMO, small concentrations of cross-linking agents (diols). These samples could not be softened and stretched by solvents or heating, and thus did not yield fibrous diffraction patterns which could provide unit cell and molecular structure information. However, they did display a limited number of powder rings in their x-ray diffraction patterns, which was an encouraging indication that micro-domains of crystalline ordering existed even in the elastic cross-linked form of the polymer.

Samples of linear $(\text{BAMO})_n$ polymers of M.W. 20,000 were later made available which were produced without the addition of cross-linking agents. Initial treatment of these samples centered on a search for a method for softening (or dissolving) these polymers in a solvent, with the aim of either stretching, re-depositing, or extruding the softened polymer in a more ordered crystalline form. Although these attempts did not succeed in their aim, the presence of a cyclic tetramer side-product was discovered, and led to the characterization of the molecular structure of the tetramer, which is a close analog to the actual polymer.

A breakthrough in the analysis of the linear polymer occurred when it was discovered that $(\text{BAMO})_n$ could be quickly melted and stretched by immersion in hot (ca $90^\circ\text{C}.$) water without any sign of decomposition. (Slower heating in air, or more extended heating did lead to slight darkening of the substance and no improvement in molecular orientation). X-ray diffraction films of this stretched sample display a characteristic x-ray fiber pattern (see Fig. 5). This pattern consists of ca. 20 extended, but distinct, spots which are much stronger, relative to background, than the powder rings observed with unstretched $(\text{BAMO})_n$.

From layer lines and spots along the stretch direction of the polymer, it is possible to immediately determine the fiber repeat distance of the polymer; i.e., the distance between exact repeats of the polymer units along the chain direction. This was found to be 4.75 \AA ; this short repeat unit is only consistent with a non-helical planar zig-zag conformation in the $(\text{BAMO})_n$ backbone.

Spots along the equator (the line perpendicular to the stretch direction) reveal the other dimensions of the crystalline unit cell, which correspond to chain-chain packing approaches. However, this information can only be obtained from a fiber pattern by indexing a few of the equatorial spots in a self-consistent manner, using the indices and the accurate locations of the peaks to calculate a set of cell dimensions, and then checking that this cell

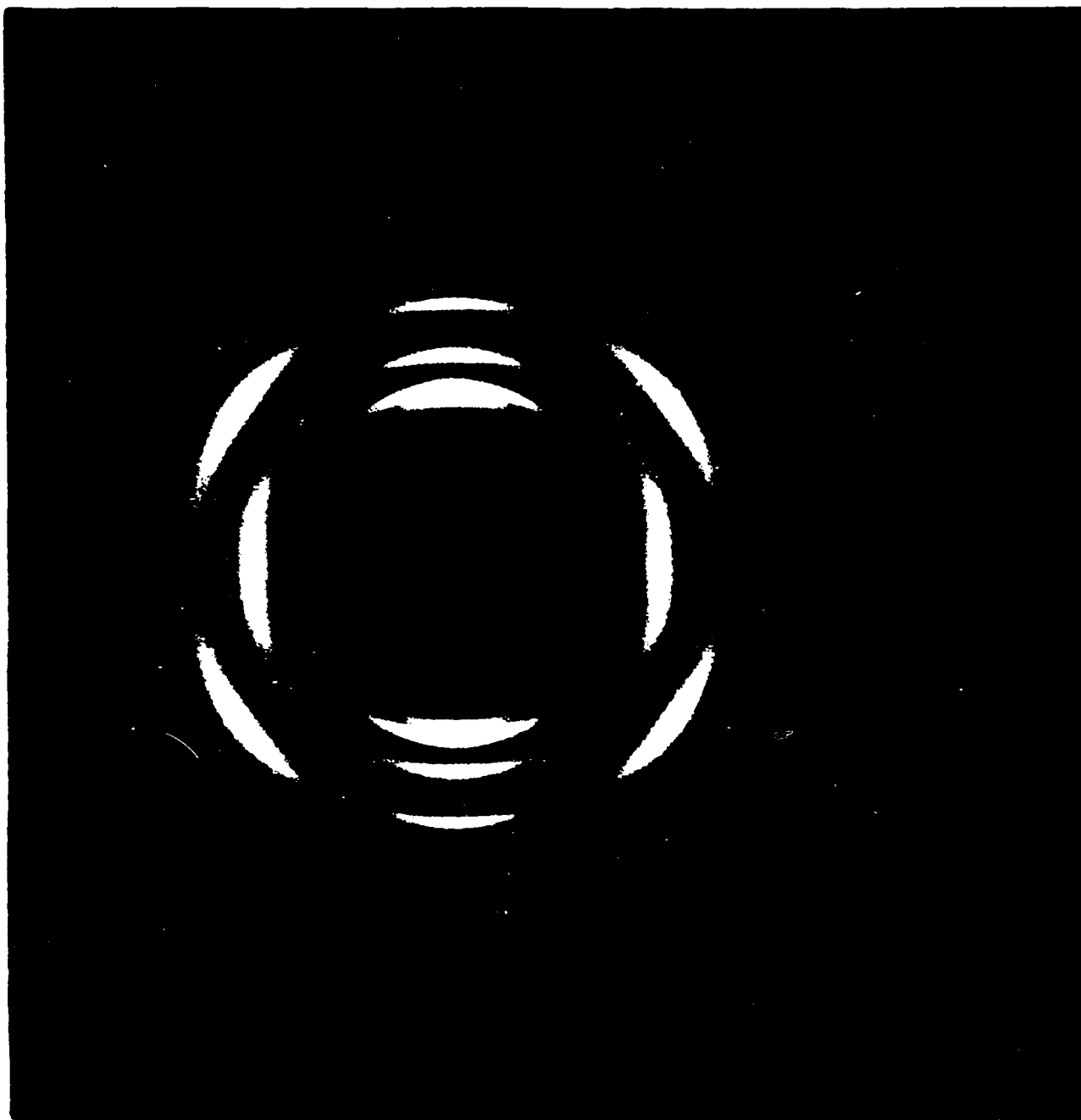


Fig. (5) The fiber x-ray diffraction pattern from stretched polymers of BAMO. The stretch direction is horizontal in this illustration. The many small spots in this photograph are diffraction spots from small crystals of the BAMO tetramer (an impurity in this polymer sample).

correctly predicts the location of all the other spots. A computer program was written to go through such trials in a systematic way. Over 20,000 indexing trials were quickly evaluated, and one orthorhombic cell fit the pattern better than all others. The cell thus determined for $(\text{BAMO})_n$ was $a = 5.77$, $b = 15.01$, and $c = 4.75 \text{ \AA}$. The calculated density from this cell is 1.356 g/cm^3 , which compares well with the tetramer density (1.336 g/cm^3), and a measured density of 1.30 g/cm^3 (from the report of Miller et al., 1981; probably refers to a cross-linked polymer sample).

All of the many components necessary for building a reasonable full crystal structure for $(\text{BAMO})_n$ have now been determined by our preliminary studies and may be systematically assembled by our computer modelling programs. The various types of information used are listed below.

1. Methylazido distances and angles - from BAMO monomer electron diffraction study
2. Backbone distances and angles - from BAMO tetramer x-ray study
3. Backbone torsions - fixed near 180° by short fiber repeat distance
4. Side-chain torsions - many possible starting values examined and ranked by non-bonded energy calculations ("Molecular Mechanics" Programs)
5. Packing - many aspects of packing are approximately fixed by size of cell and shape of chain; extinction along o_k (strongly suggested by pattern) required half-cell translational operation along y

Two criteria are being used to recognize the true crystal structure; the R-factor ($R = \Sigma |F_o - F_c| / \Sigma F_o$), which expresses the average disagreement between the observed spot intensities and the model's calculated spot intensities, and the non-bonded contact energy, which rises rapidly if adjacent molecules make contacts which are less than accepted van der Waals contact distances.

A number of fully ordered crystal structure models have been assembled, tested, and refined with the LALS (linked-atom least squares) program. Two promising models are depicted in Figs. 6 and 7. Packing model I gives a low R-factor of 18%, but it contains some short azido-azido contacts of 2.5-2.6 Å, which are too small. (The tetramer crystal had none less than 3.1 Å). Packing model II has better contacts (N...N minimum 2.9 Å), but gives a higher R-factor (ca. 35%). A better fit to both criteria is anticipated with the aid of an interactive computer graphics system which is now available. At this point, many features of the polymer's structure are emerging.

The cell geometry requires the same zig-zag backbone for the chain in all models. This planar backbone always lies in or near the AC plane. Another layer of chain backbones lies at $Y = 1/2$, giving the *oko* extinction observed in the data. The methylazido chains extend into and fill in the interlayer space. Detailed models differ only in the conformation of these side-chains, and the relative translation of the AC layers.

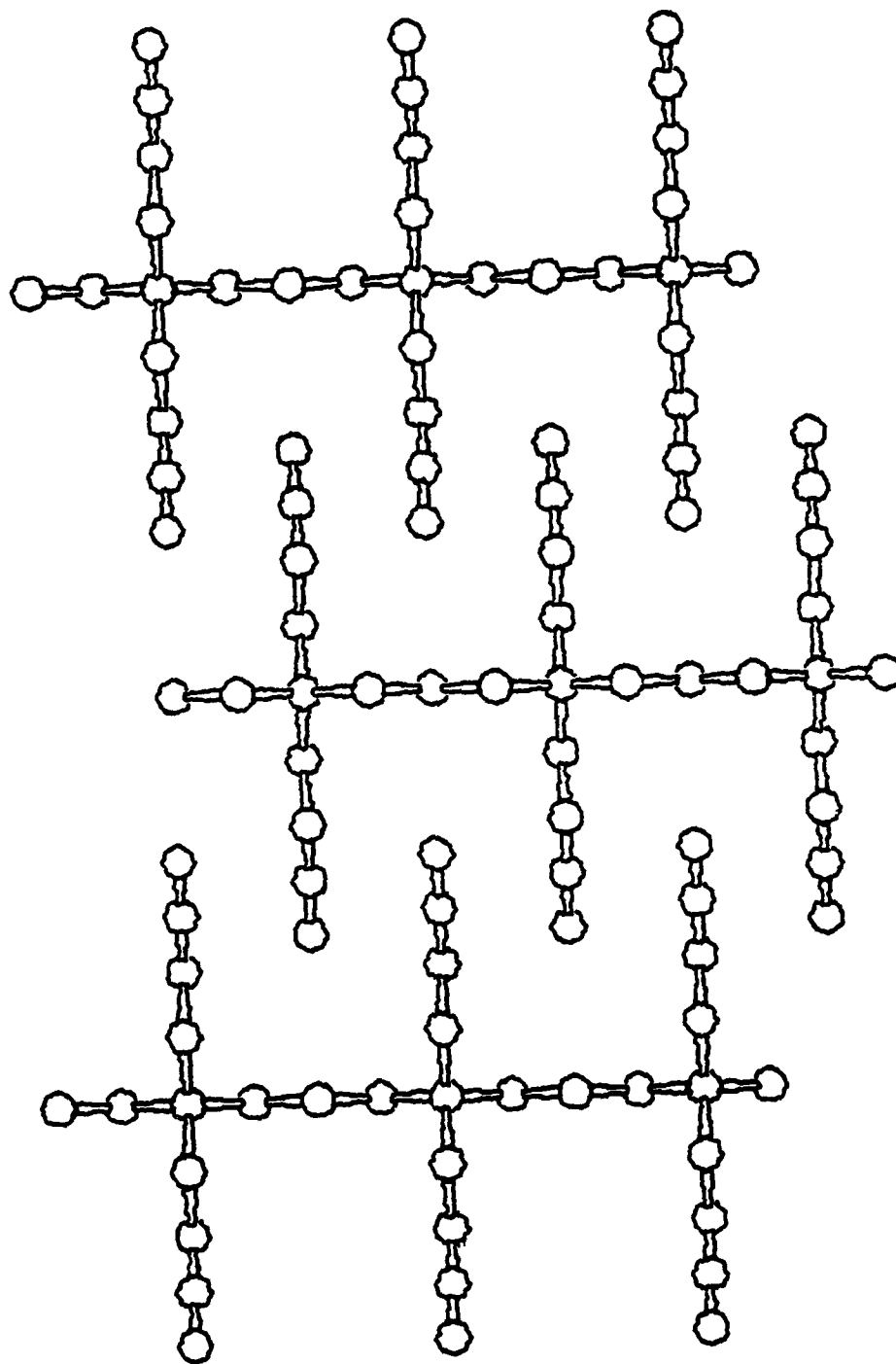


Fig. (6) Packing model I for $(\text{BAMO})_n$.

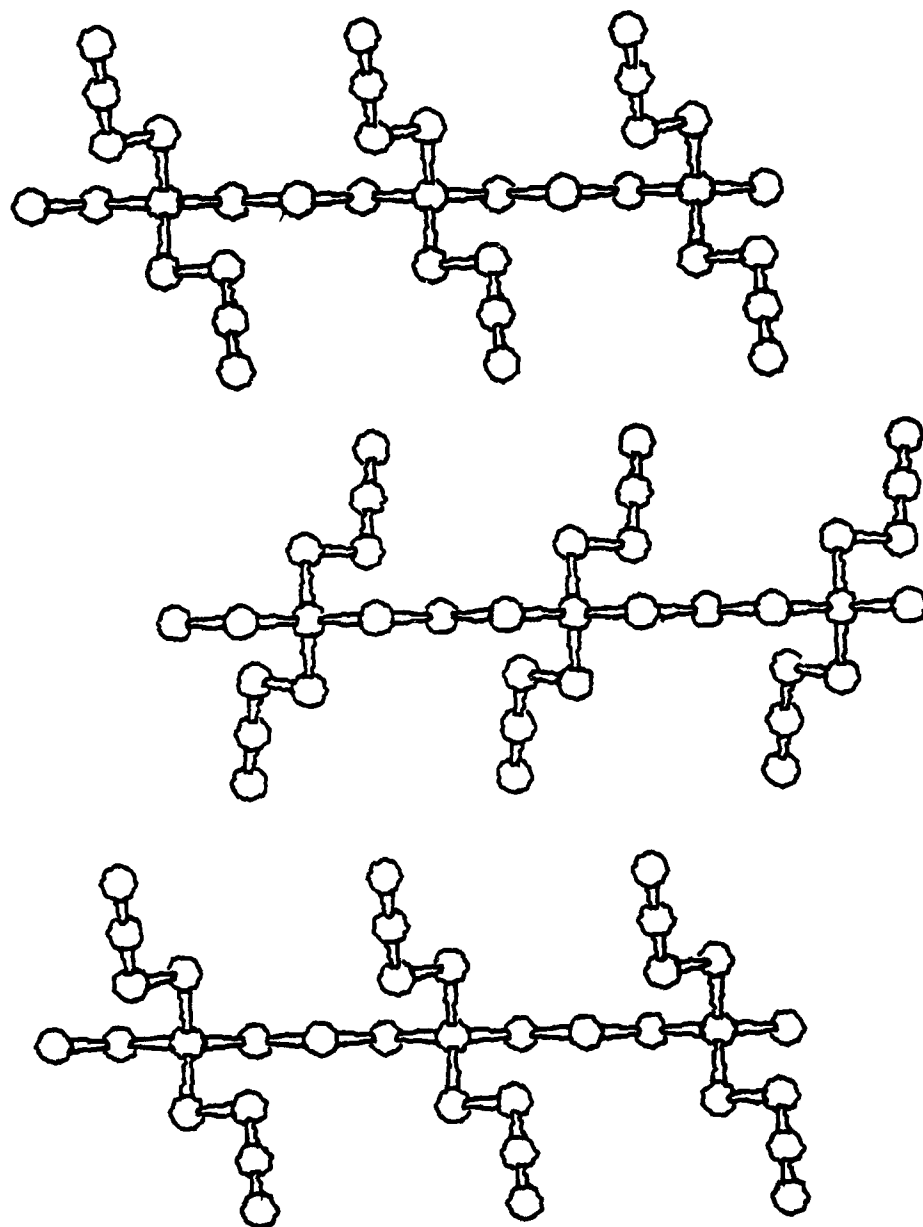
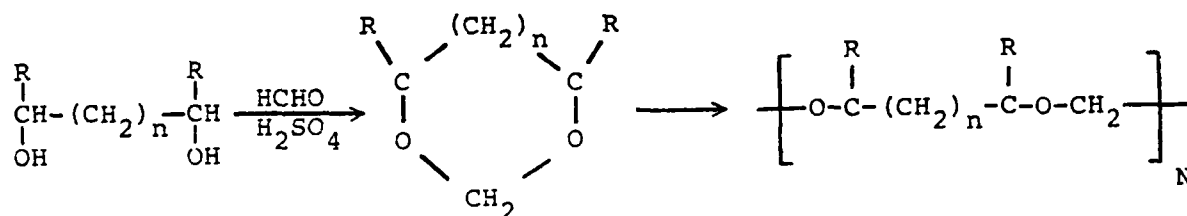


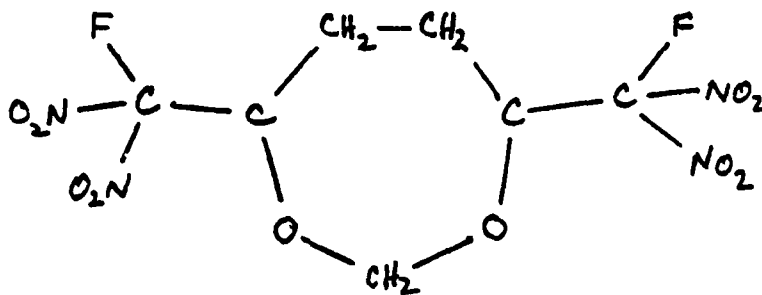
Fig. (7) Packing model II for (BAMO)_n.

V. The Crystal Structure of a Tetranitrodifluoro Monomer:
4,7-bis(Dinitrofluoromethyl)-1,3-dioxepane

A possible route for the synthesis of dense energetic polymers suggested by Dr. Horst Adolph (Naval Surface Weapons Center, White Oak) involves the preparation, and subsequent ring-opening polymerization of cyclic formals:



R is chosen to be an energetic substituent. Dr. Adolph provided crystals of one such monomer for structural analysis, a tetranitrodifluoro compound:



This compound is quite stable despite its high degree of nitro-substitution, and forms clear colorless crystals. The unit cell parameters are $a = 14.306(2)$, $b = 11.364(2)$, $c = 10.854(2)$ Å, $\beta = 128.93(2)^\circ$ and the crystal space group symmetry is $C2/c$. The crystal density calculated from this cell is 1.675 g/cm^3 .

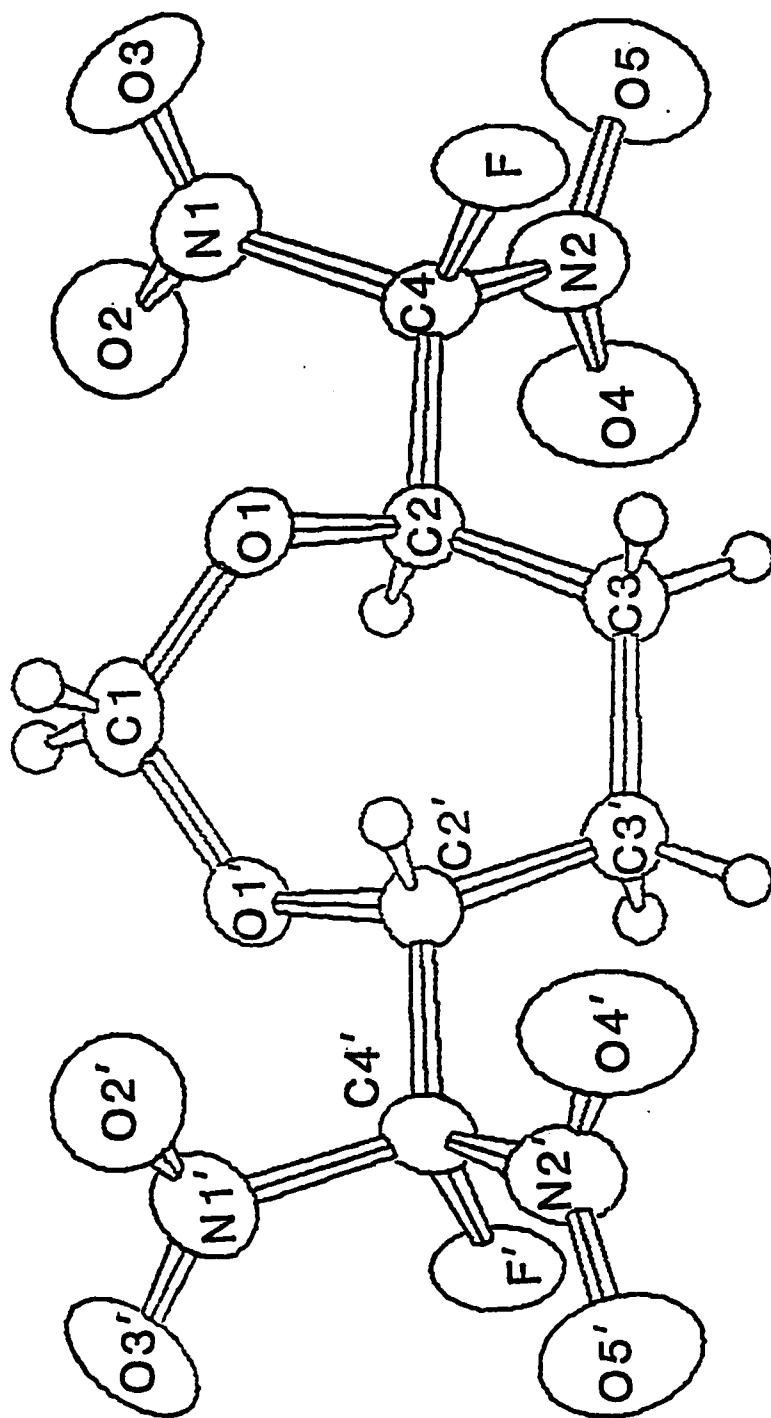


Fig. (8) The molecular structure of 4,7-bis(dinitrofluoromethyl)-1,3-dioxepane. This computer-generated perspective view is an exact representation of the refined atomic coordinates of the molecule from the diffraction analysis.

Table 3. Bond Distances, Angles, and Selected Torsions
for 4,7-bis(Dinitrofluoromethyl)-1,3-dioxepane

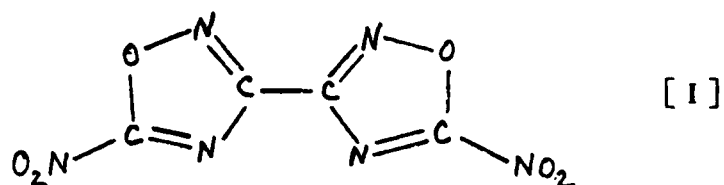
Distances (Å.)		Angles (°)	
C(1)-O(1)	1.405(4)	O(1)-C(1)-O(1)'	111.9(5)
O(1)-C(2)	1.430(5)	C(1)-O(1)-C(2)	114.3(3)
C(2)-C(3)	1.522(6)	O(1)-C(2)-C(4)	104.3(3)
C(3)-C(3)'	1.531(10)	O(1)-C(2)-C(3)	108.8(3)
C(2)-C(4)	1.495(7)	C(4)-C(2)-C(3)	113.1(4)
C(4)-F	1.333(5)	C(2)-C(4)-F	113.9(4)
C(4)-N(1)	1.530(6)	C(2)-C(4)-N(1)	109.5(4)
N(1)-O(2)	1.181(5)	C(2)-C(4)-N(2)	115.1(4)
N(1)-O(3)	1.228(6)	F -C(4)-N(1)	106.8(4)
C(4)-N(2)	1.517(6)	F -C(4)-N(2)	105.7(4)
N(2)-O(4)	1.153(5)	N(1)-C(4)-N(2)	105.2(3)
N(2)-O(5)	1.201(6)	C(2)-C(3)-C(3)'	112.8(3)
Torsions(°)		C(4)-N(1)-O(2)	115.6(5)
		C(4)-N(1)-O(3)	116.0(5)
O(1)'-C(1)-O(1)-C(2)	45.9(2)	O(2)-N(1)-O(3)	128.3(5)
C(1)-O(1)-C(2)-C(3)	-97.6(4)	C(4)-N(2)-O(4)	118.1(5)
O(1)-C(2)-C(3)-C(3)'	70.7(6)	C(4)-N(2)-O(5)	116.0(5)
C(2)-C(3)-C(3)'-C(2)'	-49.8(8)	O(4)-N(2)-O(5)	125.8(6)
O(1)-C(2)-C(4)-F	70.6(4)		
F -C(4)-N(1)-O(3)	-3.5(6)		
F -C(4)-N(2)-O(5)	-33.9(6)		

The crystal and molecular structures were obtained by routine application of the symbolic addition procedure (Karle and Karle, 1966). Bond distances and angles for this molecule are listed in Table 3, and a graphic representation of the molecule is illustrated in Fig. 8. This structure appears to be the first ever reported of a compound containing a dinitro-carbon atom. Only two prior reports have so far been found dealing with mono-nitro substitution on tetrahedral carbon atoms.

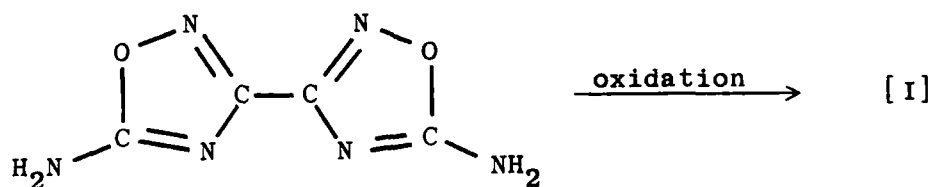
The results of this study were transmitted to Dr. Adolph and also to Dr. Joyce Kaufman (Johns Hopkins Univ.) for quantum chemical analysis of the electrostatic potential and reactivity of this monomer. A short article is being prepared for publication in *Acta Crystallographica*.

VI. The Crystal Structure of a Diaminodioxadiazole, a Synthetic Intermediate Originating in Research on the Synthesis of New High Explosives

The compound bis(5-nitro-1,2,4-oxadiazole),

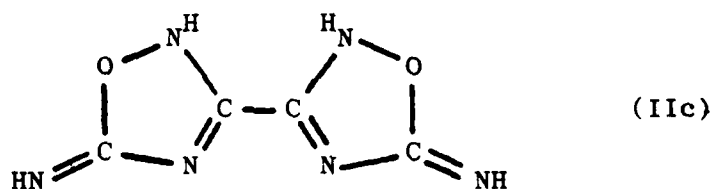
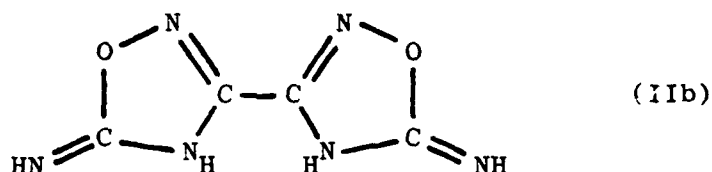
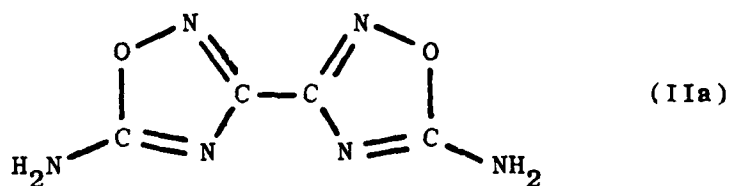


has been proposed as a possibly useful high density explosive (C. Coon, 1981). However, synthesis by direct nitration of the unsubstituted dioxadiazole did not proceed, even under harsh conditions. An alternative synthetic route would be via the synthetic intermediate II, a diaminodioxadiazole:



Compound II was synthesized by Dr. Coon, but the above reaction also did not proceed, even with very strong oxidizers. An x-ray diffraction study of II was proposed to establish the

nature of the bonding in the starting material. A number of structures might be possible which differ only in the placement of the hydrogen atoms:



A sample of II was obtained from Dr. Coon and analyzed by means of x-ray single-crystal diffraction. The results unambiguously locate the hydrogen atoms, and corroborate that the structure is IIa. We are informed that this is also consistent with a ^{13}C -NMR spectral study. Bond distances and angles of II are displayed in Fig. 9. The extraordinary stability of this compound may be reflected in the detailed geometry of the amino group. The entire molecule, including the amino hydrogen atoms, is planar. This fact, coupled

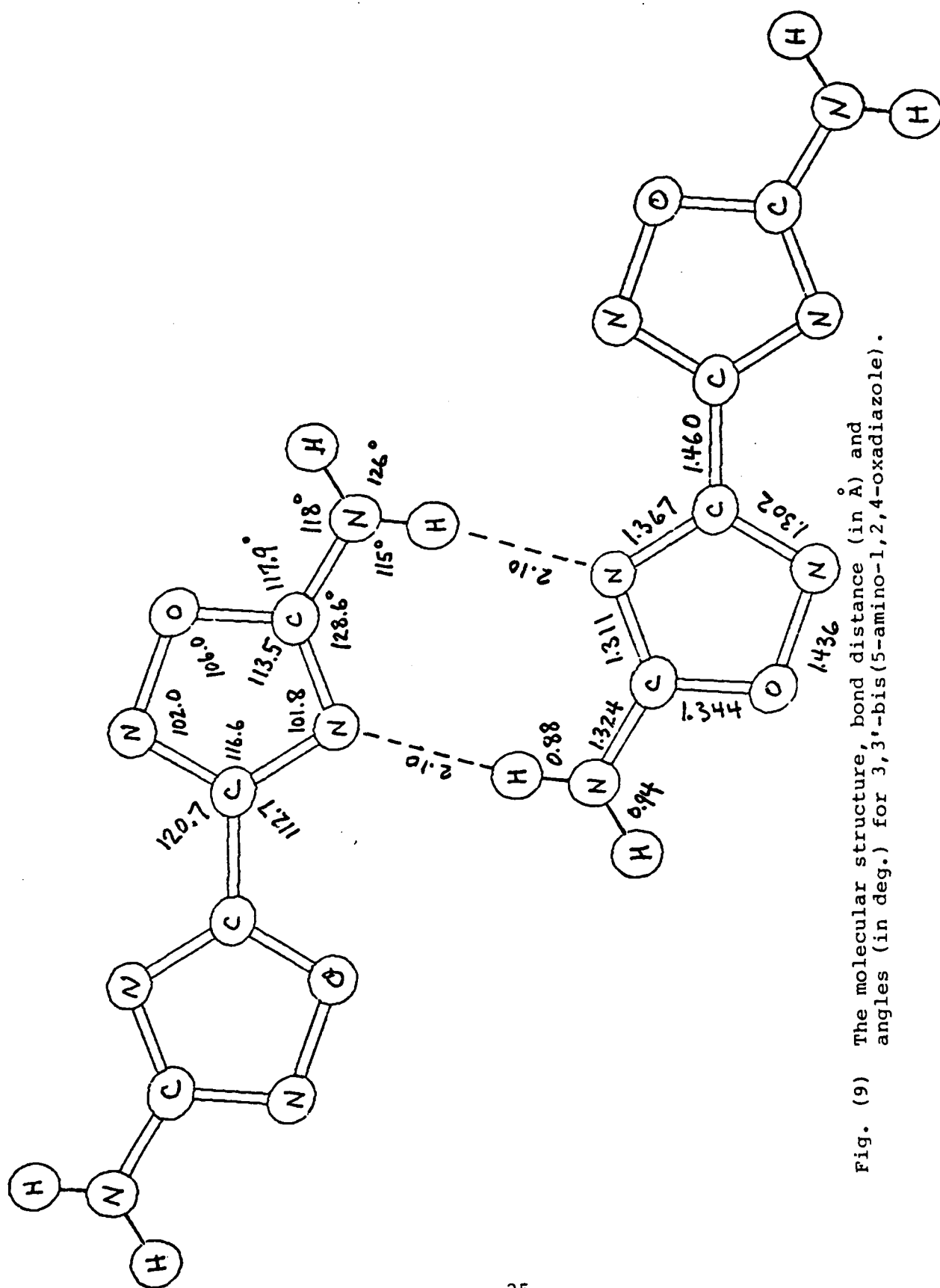


Fig. (9) The molecular structure, bond distance (in Å) and angles (in deg.) for 3,3'-bis(5-amino-1,2,4-oxadiazole).

with the very short length (1.324 Å) of the CN "single" bond, indicates extensive conjugation between the ring electrons and the amino lone-pair electrons. Thus, this amine bears little resemblance to a primary aliphatic amine. Structurally, it is related to the aromatic amines or to the amino portion of an amide group. Another class of compounds which contain similar conjugated exocyclic amino groups are the pyrimidines. This information was transmitted to Dr. Coon, in hopes that it might aid in guiding future efforts towards the synthesis of I.

VI. References

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